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**MULTI-WALL CARBON
NANOTUBES FOR FLOW-INDUCED
VOLTAGE GENERATION (Preprint)**

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Multi-Wall Carbon Nanotubes for Flow-Induced Voltage Generation

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Abstract

Recently it has been reported that voltage can be generated by passing fluids over single-walled carbon nanotube (SWCNT) arrays with potential application to flow sensors with a large dynamic range. The present work investigates voltage generation properties of multi-walled carbon nanotubes (MWCNT) as a function of the relative orientation of the nanotube array with respect to the flow direction, flow velocity, and solution ionic strength. It was found that the flow-induced voltage can be significantly enhanced by aligning the nanotubes along the flow direction, increasing the flow velocity and/or the ionic strength of the flowing liquid. A flow-induced voltage of ~ 30 mV has been generated from our perpendicularly-aligned MWCNT in an aqueous solution of 1 M NaCl at a relatively low flow velocity of 0.0005 m/s which is 15 times higher than the highest voltage reported for single-wall carbon nanotubes. The results are generally consistent with the pulsating asymmetric ratcheting mechanism proposed for SWCNT arrays, in which an asymmetrical spatial distributed strain forms from interactions with the polar and ionic species at the tube surface and is driven along the tube by the fluid flow.

Keywords: Aligned carbon nanotubes; Flow Sensor; Patterning; Flow-Induced Voltage.

I . INTRODUCTION

The excellent electronic^{1,2} and mechanical properties of carbon nanotubes,^{3,4} together with their unique molecular structure,⁵ have made them very attractive for many potential applications including nano-scale electronics,^{6,7} actuators,⁸ and sensors.⁹ Of particular interest is the recent theoretical investigation of electrical current generation from the immersion of a metallic single-wall carbon nanotubes (SWCNTs) in a flowing liquid.¹⁰ One proposed mechanism is based on “pulsating asymmetric ratcheting” in which a spatial-distributed strain forms due to interactions between the polar and ionic species of the flowing fluid and the tube surface.¹¹ It has been suggested that the radial and axial nanotube strain induced by the fluid interactions are primarily due to electrostatic interactions of the fluid species with the delocalized π electrons of the nanotube and not from charge transfer between the ions of the fluid and the nanotube.¹² Alternative proposed mechanisms involve either electrons released by thermal excitation with the hot phonons produced by the friction of the moving liquid or the direct scattering of free carriers from the fluctuating Coulombic fields of the ions or polar molecules in the flowing liquid.¹⁰

In this case of the pulsating ratcheting mechanism, an asymmetric potential drags carriers along the tubes in the direction of the flow based on the sense and magnitude of the asymmetry to give a non-linear dependence on flow velocity. A symmetric potential can also drag carriers, but will demonstrate a linear dependence of the induced voltage on flow velocity. The dominating sense of the bias is related to the details of the electrostatic interactions with the fluid and can be probed by varying the contents of the fluid. The magnitude of the asymmetry is thought to be related to the flow velocity (u) via the shear-deformed velocity gradient that is established at the nanotube surface. The non-linear voltage dependence of an individual

nanotube in a specific fluid can be mathematically expressed for small degrees of asymmetry in terms of exponential fitted parameters (V_o , a & b) and compared by the following expression¹³

$$V \cong \frac{V_o(1 - e^{-a u})}{e^{-b u}} \quad (1)$$

Although more investigation is still needed to clarify the mechanism of the flow-induced voltage generation, many of the above-mentioned dependences for the pulsating asymmetric ratcheting mechanism have been experimentally observed. For example, Sood and his co-workers¹³ have recently reported that the flow of liquids such as water, methanol, water-glycerol mixtures, and HCl solutions on single-walled carbon nanotube bundles induces a voltage in the sample along the direction of the flow that varies proportionally with the log of the liquid flow velocity over nearly six decades of velocity. These authors attributed this highly nonlinear voltage-velocity response to the aforementioned pulsating asymmetric ratcheting mechanism, though the possible effects associated with the nature of the fluid-tube interactions and the packing or morphology of the nanotubes have yet been fully investigated. Ensembles of connected nanotubes such as those in nanotube arrays present a complication in that currents and voltages produced in individual tubes can interact to obscure the underlying physics. Thus, the flow-induced charge generated will be dependent on the nature of the fluid-tube interactions, the flow direction and velocity, as well as the packing or morphology of the nanotube arrays. These parameters will be investigated for the CVD grown MWCNT arrays.

We have previously developed a simple pyrolytic method for large-scale production of aligned carbon nanotube arrays *perpendicular* to the substrate.¹⁴ Meanwhile, we have used photolithographic and soft-lithographic techniques for patterning the aligned carbon nanotubes with a sub-micrometer resolution.^{15, 16} These aligned carbon nanotube arrays can be transferred onto various substrates of

particular interest in either a patterned or non-patterned fashion.¹⁷ This, coupled with their nonaligned counterparts and surface functionalization techniques, opens many possibilities for investigating the properties and applicability of charge generation with multi-walled carbon nanotubes (MWCNTs). In this paper, we report, for the first time, the flow-induced voltage generation for MWCNTs. Using MWCNTs with diverse morphological structures, including nonaligned and aligned forms, we have studied the effects of the nature of the fluid-tube interactions, the flow direction and velocity, and the packing or morphology of the nanotubes on the flow-induced voltage for MWCNTs. Our results are consistent with the proposed pulsating asymmetric ratcheting mechanism for the flow-induced voltage generation of MWCNTs.

II . EXPERIMENT

Materials. Carbon nanotubes used in this study were prepared by the pyrolysis of metal-organic complexes,¹⁸ and metal-catalysed reactions,¹⁹ which produces both nonaligned and aligned MWCNTs of controllable tube length. For horizontally-aligned carbon nanotubes, the substrate-supported layers of metal catalyst were prepared from 0.006 M solutions of $\text{Fe}(\text{NO})_3 \cdot 9\text{H}_2\text{O}$ in acetone. After drying, the substrates were introduced in a furnace under Ar/H_2 at 750 °C for 5 min (calcination), followed by CVD growth of carbon nanotubes on the substrate in a quartz tube furnace under a combined flow of Ar (600 sccm), H_2 (15 sccm), and C_2H_2 (10 sccm) at 900 °C for 10 min.¹⁹ The nonaligned MWCNTs were grown from metal-organic chemical vapor deposition involving immersing a quartz plate in the same iron colloid solution for 20 min and pre-heating in a quartz tube furnace under Ar/H_2 at 800 °C for 5 min and followed by growth of MWCNTs on the substrate under a combined flow of Ar (600 sccm), H_2 (20 sccm), and C_2H_2 (20 sccm) for 20 min.²⁰ The perpendicularly-aligned MWCNTs were produced by pre-heating a quartz plate in a quartz tube furnace under Ar/H_2 at 800 °C for 5 min,

followed by continuously injecting 3-10 mL (1 mL/min) xylene/ferrocene for 3-10 min under a combined flow of Ar (400 sccm)/H₂ (20 sccm) at 800 °C.¹⁸

Measurements of the flow-induced voltage. The *as-synthesized* carbon nanotubes were removed from the quartz substrate in an aqueous HF, as previously reported.¹¹ All of the three different types of carbon nanotube films (*i.e.* nonaligned carbon nanotubes, horizontally-aligned carbon nanotubes, and perpendicularly-aligned carbon nanotubes) were used to prepare the samples for the flow-induced voltage generation and subsequent measurements, as shown in the Figure 1.

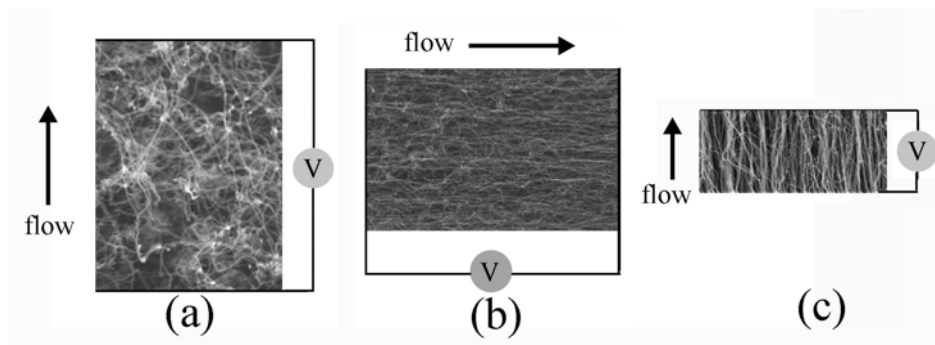


FIG. 1 Carbon nanotube samples for the flow-induced voltage measurements: (a) nonaligned carbon nanotubes, (b) horizontally-aligned carbon nanotubes, and (c) perpendicularly-aligned carbon nanotubes.

Figure 2 schematically shows the experimental setup used for measuring the flow-induced voltage for the MWCNT samples prepared above, which were soaked in pure water for 2 hours prior to the measurement. As can be seen in Figure 2, the carbon nanotube sample was then placed at the center of a relatively large cylindrical plastic flow chamber (Part b of Figure 2) to avoid any turbulent flow that may have been caused by the expansion of the flow at the inlet of the flow chamber. The flow velocity crossing the carbon nanotube sample was measured and monitored by a digitized valve for the bulk flow (Part a of Figure 2). The flow-induced voltage on the nanotube sample along the flow direction was measured by a FLUKE 45 Dual Display multimeter (Part c of Figure 2), which was connected to a

computer for data acquisition (Part d of Figure 2).

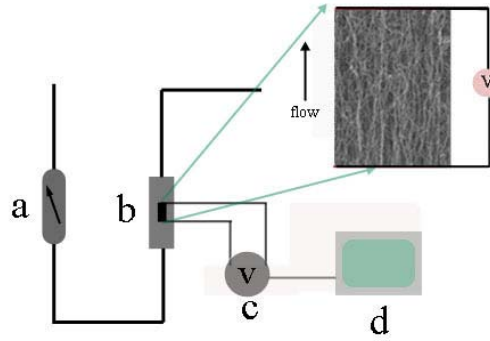


FIG. 2 Schematic illustration of the experimental setup for detecting the flow-induced voltage for carbon nanotubes: (a) a digitized valve for controlling and measuring the flow velocity; (b) a cylindrical plastic flow chamber; (c) a multimeter for detecting the flow-induced voltage; (d) a computer for data acquisition.

III. RESULTS AND DISCUSSION

The effects of the flow velocity and the nanotube packing/morphology. To investigate the influence of the packing/morphology of carbon nanotubes on the flow-induced voltage generation, we used nonaligned, horizontally-aligned, and perpendicularly-aligned MWCNTs. Figure 3 shows typical SEM images for all of the three different types of MWCNTs used in this study. As can be seen in Figures 3a and 3b, the densely-packed nonaligned (disordered) MWCNTs were formed on the quartz plate with a low surface density of the iron catalyst nanoparticles. The horizontally-aligned carbon nanotubes with orientation along the surface are shown in Figure 3c and 3d. Figures 3e and 3f reveals the perpendicularly well-aligned carbon nanotubes with a uniform length of about 70 micrometer. The outer diameter of the tubes are consistent for the different morphologies and on the order of *ca.*20 nm

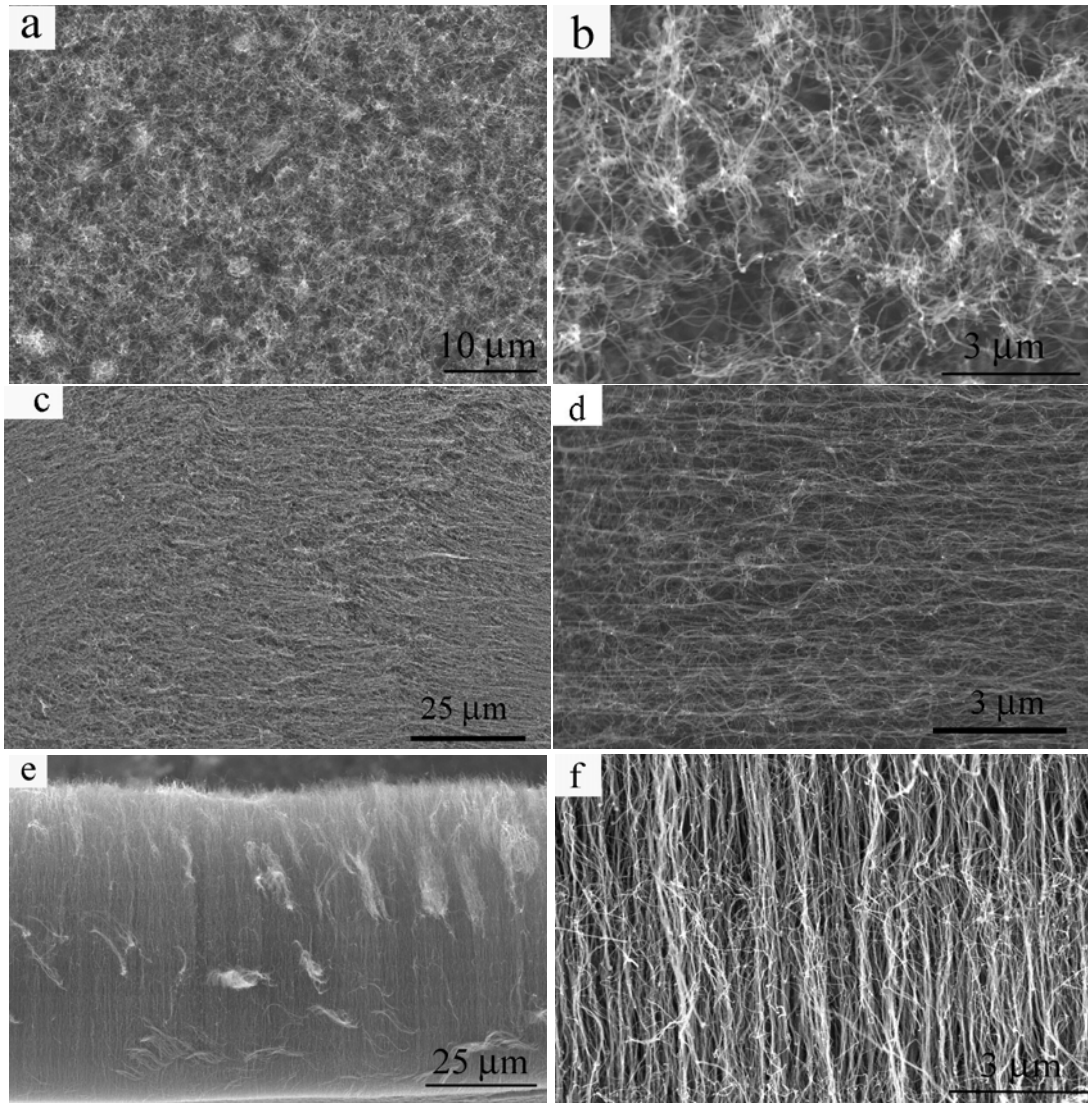


FIG. 3 SEM images of disorder carbon nanotubes under (a) a low magnification view and (b) a high magnification; SEM images of horizontally-aligned carbon nanotubes under (c) a low magnification and (d) a high magnification; and SEM images of perpendicularly-aligned carbon nanotubes under (e) a low magnification and (f) a high magnification.

For the measurements of the flow-induced voltages, we fabricated nanotube films with varying dimension depending on the sample type and measured the voltage response with flow. For example, the non-aligned sample film was 3 cm long, 2 cm wide, and approximately 10 μm thick and was measured with the flow passing across the 3 cm dimension. The horizontally-aligned nanotube film was also 3 cm long, 2 cm wide and measured with the flow passing across

the 3 cm dimension. The perpendicular-aligned nanotube film was 2 cm long, 0.5 cm wide and 70 μm thick and measured with flow applied from directly above the film similar to a filtering mechanism. Each of the samples has approximately the same amount of carbon nanotubes (by weight) within a similar volume. However, the full flow velocity that is experienced at the surface of the nanotube films in the direction of the flow is approximately 6 times larger for the non-aligned and horizontally aligned films (3 cm x 2 cm surface) than for the vertically aligned film (2 cm x 0.5 cm surface). Flow velocities below this surface are more difficult to predict especially as back flow occurs at higher velocities.

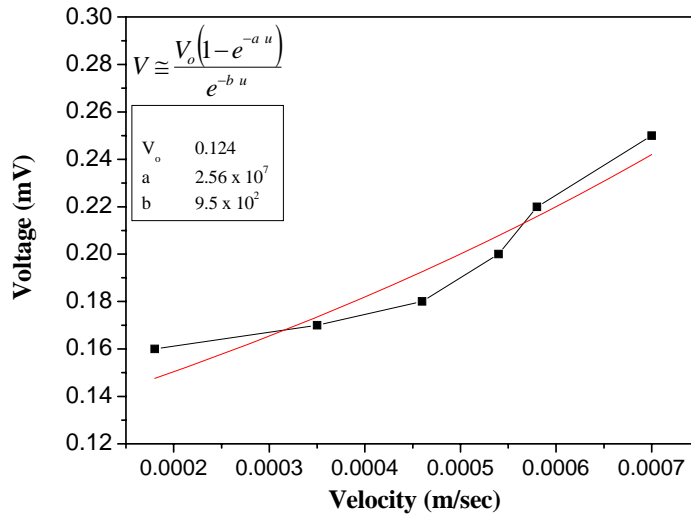


FIG. 4 Variation of the flow-induced voltage with the flow velocity of water for nonaligned MWCNTs

Figure 4a shows the dependence of the flow-induced voltage on the flow velocity of pure water for non-aligned carbon nanotubes. It can be seen clearly that the flow-induced voltage does vary with the flow velocity. At a flow velocity of 0.0005 m/s, a voltage of ~0.2 mV was generated, which is about 10 times lower than the corresponding voltage generated by SWCNTs.⁸ By plotting the flow-induced voltages against the flow velocities, a non-linear relationship was obtained, as shown in Figure 4. The red line in the Figure 4 shows the fit of our

data for the functional form in Eq. 1 with $V_o=0.12$, $a=2.56 \times 10^7$, and $b=9.50 \times 10^2$. The voltage generated along MWCNTs is about 10 times smaller than the voltage produced by the single-walled carbon nanotubes for similar dimensions and flow velocities.

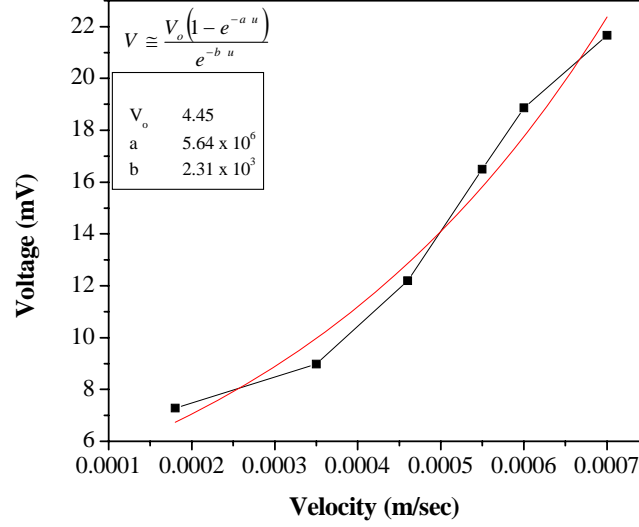


FIG. 5 Variation of the flow-induced voltage with the flow velocity of water for the horizontally-aligned MWCNTs

To investigate the effect of the nanotube packing/morphology on the flow-induced voltage, we proceeded with the horizontally-aligned carbon nanotubes. As can be seen in Figures 5, the dependence of the flow-induced voltage on the flow velocity of pure water for the horizontally-aligned carbon nanotubes takes a similar course to that of the nonaligned carbon nanotubes (*cf.* Figure 4). However, relatively higher flow-induced voltages were obtained for the former at any fixed flow velocity of pure water. For comparison, a voltage of ~14 mV was generated by the horizontally-aligned carbon nanotube sample at the flow velocity of 0.0005 m/s, which is about 70 times larger than the corresponding voltage produced by the nonaligned carbon nanotubes under the same condition. The red line in the Figure 5 shows the fit of our data for the functional form in Eq. 1 with $V_o=4.45$, $a=5.64 \times 10^6$ and $b=2.31 \times 10^3$. The relatively high voltage observed

for the horizontally-aligned MWCNTs is attributable to a *collective* (additive) response in the flow-induced voltage from the alignment of a substantial number of individual nanotubes along the flow direction.

Having demonstrated the flow-induced voltage generation for nonaligned and horizontally-aligned MWCNTs, we further investigated the perpendicularly-aligned MWCNTs. Figure 6a shows changes of the flow-induced voltage with the flow velocity of pure water. Compared with its non perpendicularly-aligned counterpart, the perpendicularly-aligned MWCNT sample produced a higher voltage at a fixed flow velocity of water within the velocity range covered by this study. For instance, a voltage of ~ 12.5 mV was generated at the flow velocity of 0.0005 m/s, which is about sixty and six times higher than the voltages produced by the nonaligned carbon nanotubes and SWCNTs,⁸ respectively, at the same flow velocity of pure water. The relatively high voltage observed for the perpendicularly-aligned MWCNTs is attributable to the fact that each of the constituent nanotubes aligns in the same direction towards the water flow, leading to a maximized *collective* response in the flow-induced voltage. Unlike the nonaligned MWCNTs, we also found that the perpendicularly-aligned MWCNT sample showed a decrease in the flow-induced voltage at a flow velocity of pure water above ~ 0.0005 m/s. The dependence of the flow-induced voltage on the flow velocity over the entire range of the flow velocity covered in this study is given in Figure 6b, which shows a rapid increase in the flow-induced voltage within the range of relatively low flow velocities (> 0.0004 m/s). The observed reverse proportion of the flow-induced voltage to the flow velocity at high flow velocities is, most probably, due to the occurrence of significant water refluent (back flow) along the aligned carbon nanotubes at high flow velocities, as one side of the aligned nanotube sample film was sealed by a supporting substrate (*e.g.* plastic film) to enhance its mechanical strength (see, Figure 1). Thus, the actually velocity of the flow is changed in magnitude and possibly direction from the values given by the

flow meter and the previously observed non-linear voltage behavior is not observed, and hence these data points were not fitted to the given expression. Although the exact value of the flow-induced voltage for aligned perpendicularly MWCNTs may vary somewhat from sample to sample, depending the nanotube length, diameter, and packing density, the above results suggest that relatively high flow-induced voltages can be generated from aligned carbon nanotube arrays by optimizing the nanotube structure and sample fabrication.

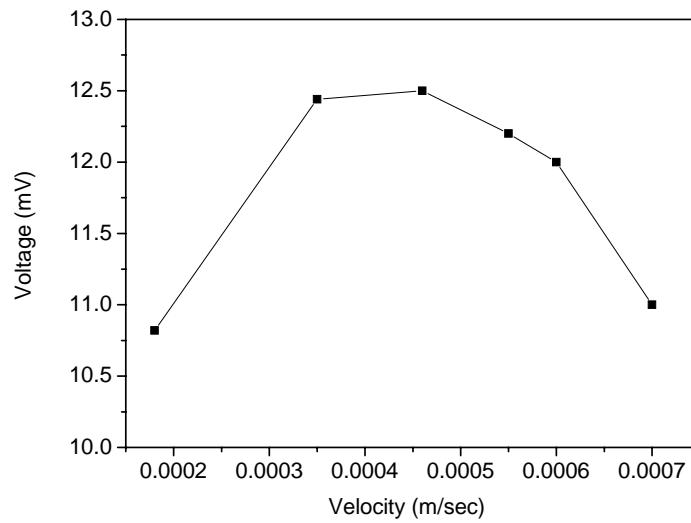


FIG. 6 Variation of the flow-induced voltage with the flow velocity of water for perpendicularly - aligned MWCNTs

The effects of the nature of the fluid-tube interactions. To investigate the effects of the nature of the fluid-tube interactions on the flow-induced voltage, we used various aqueous solutions of NaCl with different salt concentrations to regulate interactions of the nanotube surface with the ionic species in the fluid flow. Figures 7a show the dependence of the flow-induced voltage on the flow velocity of the NaCl solutions with different salt concentrations for the horizontally-aligned carbon nanotubes. The flow-induced voltages increase with increasing the NaCl concentration at a fixed flow velocity. The dependence of the flow-induced voltage on

the NaCl solution at the fixed flow velocity of 0.0005 m/s is given in Figure 7b, which shows a rapid increase in the flow-induced voltage at the low NaCl range, followed by a retarding of the increase rate in the flow-induced voltage with increasing the NaCl concentration. The ultimate value of ~ 29 mV obtained at flow velocity of 0.0005 m/s in this particular case for the horizontally-aligned carbon nanotubes is two times higher than that for pure water, confirming the strong influence of the ionic strength of the flowing liquid on the flow-induced voltage, as predicated by the pulsating asymmetric ratcheting mechanism.

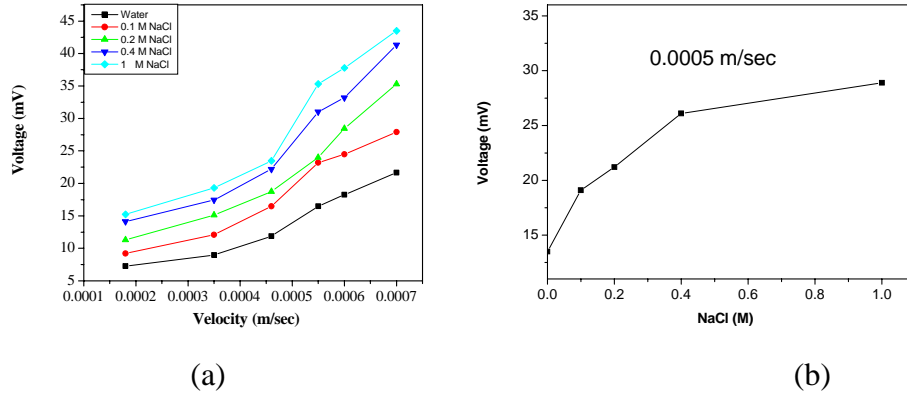


FIG. 7 (a) Variation of the flow-induced voltage with the flow velocity of the aqueous NaCl solution for the horizontally-aligned MWCNTs at different NaCl concentrations; (b) Dependence of the flow-induced voltage on the NaCl concentration at a fixed flow velocity of 0.0005 m/s.

The increase in the flow-induced voltage with increasing the salt concentration was also observed for the perpendicularly-aligned carbon nanotubes. Figures 8a and 8b show the dependence of the flow-induced voltage on the flow velocity of the NaCl solutions for a perpendicularly-aligned carbon nanotube sample. The salient feature to note in Figure 8a is that the flow-induced voltage at a fixed of flow velocity increases with increasing the NaCl concentration, though the similar trend as that shown in Figure 6a was observed for the dependence of the flow-induced voltage on the flow velocity for each of the salt solution. By plotting the flow-induced voltages at the fixed flow velocity of 0.0005 m/s against the NaCl concentrations, we obtained a linear relationship, as shown in Figure 8b, indicating that the flow-

induced voltage proportionally increases with increasing the NaCl concentration up to ~ 30 mV for the 1 M NaCl aqueous solution at the fixed flow velocity of 0.0005 m/s. These results, once again, suggest that the pulsating asymmetric ratcheting mechanism governs the interaction between the perpendicularly-aligned carbon nanotubes and the flowing liquid.

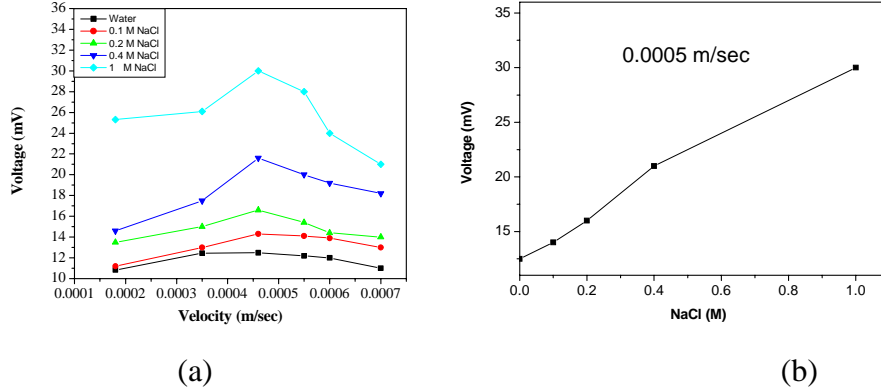


FIG. 8 (a) Variation of the flow-induced voltage with the flow velocity of the aqueous NaCl solution for the perpendicularly-aligned MWCNTs at different NaCl concentrations; (b) Dependence of the flow-induced voltage on the NaCl concentration at a fixed flow velocity of 0.0005 m/s.

IV. CONCLUSIONS

Like single-wall carbon nanotubes, we have also observed the flow-induced voltage generation for multi-wall carbon nanotubes. Our results indicate a non-linear voltage dependence on flow rate for multi-wall carbon nanotubes that is consistent with a pulsating asymmetric ratcheting mechanism proposed for single-wall carbon nanotubes.⁷ The voltage is strongly dependent on the nanotube morphology as can be observed from the two order of magnitude increase in voltage in going from the non-aligned to the vertically aligned MWCNT. For a given tube orientation, the voltage could also be more than doubled by increasing the NaCl concentration of flowing solution from 0 to 1 M. For example, a flow-induced voltage of ~ 30 mV was generated from perpendicularly-aligned MWCNT in an aqueous solution of 1 M NaCl at a relatively low flow velocity of 0.0005 m/s. This is roughly 15 times higher than the highest voltage reported for single-wall carbon nanotubes. The increase in voltage is believed to be due

the increase in charge collection efficiency of the MWCNT due to the metallic nature of the CVD produced MWCNTs. Further research in this area could lead to flow-induced nanotube voltage generators and flow sensors of practical significance.

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